



Dynamics of Photoexcitation at Nanostructured Carbon Interfaces

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Final Report

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Dynamics of Photoexcitation and Photocatalysis at Nanostructured Carbon Interfaces

Michael S. Arnold, Robert J. Hamers, and Martin T. Zanni

Department of Materials Science and Engineering and Department of Chemistry
University of Wisconsin-Madison

Abstract

During this grant, we have made major contributions to the understanding and practice of photoexcitation and photocatalysis at nanostructured carbon interfaces. There were two aspects to this grant. One aspect was to study the dynamics of films of carbon nanotubes for next generation photovoltaics and photodetectors. The other was to study the dynamics of CO₂ reduction using solvated electrons. We made excellent progress on both fronts, with each direction leading to publications in high-impact journals. With regard to the carbon nanotubes, the highlight was developing a new technique that we called two-dimensional white-light (2D WL) spectroscopy. With it, we mapped the energy flow through thin films of semiconducting carbon nanotubes with unprecedented resolution. We learned that energy does not hop between parallel tubes as one might expect from typical resonant-types of energy transfer processes, but instead occurs at the intersections between tubes, leading to dramatic changes in the direction of energy propagation. This work was published in *Nature Communications*. Regarding CO₂ reduction, we demonstrated highly selective photochemical reduction of CO₂ to CO with virtually no reduction of hydrogen (published in *Angewandte Chemie*) and have explored how to optimize this process using an understanding of the kinetics of solvated electron reactions. At moderate (10-20 atmosphere) pressures of CO₂ the rate of CO₂ reduction greatly exceeds the rate of H⁺ reduction at pH=3.5, yielding high selectivity for CO₂ reduction over H⁺ reduction. Selective reduction of CO₂ without oxidation of the diamond photocatalyst also requires providing a mechanism for a corresponding oxidation reaction, either by using a secondary Pt electrode or by including a hole scavenger such as sulfite. We have since identified conditions under which CO₂ can be also reduced to CO using inexpensive diamond nanocrystals, like that commonly used as a polishing grit.

Detailed Accomplishments:

1. Dynamics of energy transport in coupled carbon nanotube materials for next generation solar cells

In previous annual reports we summarized our experiments using pump-probe spectroscopy to probe energy transfer in thin films of electronic-type controlled semiconducting carbon nanotubes. Semiconducting carbon nanotubes are intriguing materials for photovoltaic and photodetector devices, as well as FETs and other devices, because they have strong optical absorptivity, ultrafast charge and energy transport, excellent stability, and economical solution-processability. We made excellent progress in better characterizing these exciting materials and in the synthesis of variations of these thin films. The pump-probe experiments were published in *ACS Nano* and *Nano Letters*, which are premiere journals for the nanosciences.

During the last year of this grant, we made a technological breakthrough in the way that energy transfer can be monitored in these and many other materials. The absorption spectrum of this film stretches from the visible to the near-infrared, which is a good property for light harvesting applications, but makes it difficult to study experimentally. The standard approach is to study one electronic transition at a time, using a tunable pump pulse followed by a continuum probe pulse, like we did in the first two years of our

grant. Now, we have developed a new technique that we call two-dimensional white light spectroscopy (2D WL). 2D WL spectroscopy uses a broadband continuum as the pump source that enables us to simultaneously examine a spectral range spanning from 500-1400 nm. The 2D WL spectra resolve energy transfer between all possible combinations of excitonic states in the band gap-selected nanotubes, thereby providing an instantaneous and comprehensive snapshot of the dynamical pathways. We observe exciton hopping, exciton dissociation, and anti-correlated energy levels; all of which have important implications in the development of carbon nanotube electronics and optoelectronics. This work was published in *Nature Communications*. We have also demonstrated it on quantum dots and on pentacene thin films to establish it as a general tool for measuring energy transfer. We are very excited about this technique and have been getting much attention for its implementation.

Accomplishments in synthesizing tailored coupled carbon nanotube materials and devices: We have post-synthetically isolated two populations of electronic-type sorted semiconducting carbon nanotubes, with bandgap distributions tailored for photovoltaic devices, for measurements of ultrafast photoexcitation dynamics in isolated thin films and solar cells made from coupled nanotubes. First, we have used poly(fluorene) derivatives to isolate an intentionally polydisperse mixture consisting of 5 discrete bandgaps of nanotubes ranging from 1.17 eV to 0.93 eV. Each nanotube can be spectrally resolved, allowing us to map inter-nanotube energy transfer kinetics. Second, we have isolated nearly monochiral (7, 5) nanotubes with a bandgap of 1.17 eV, allowing us to probing energy transfer kinetics without energy loss. We have learned so far how to decrease and increase inter-nanotube coupling in nanotube thin films by controlling the polymer:nanotube ratio within the films. Moreover, we have discovered how to tailor inter-tube coupling by controlling the film morphology. Towards this end, we have discovered how to create extremely porous and low density films of semiconducting nanotubes by co-casting the nanotubes with a sacrificial polymer and eliminating the polymer using a critical point drying process. This process creates aerogel structures that are 99.8% porous. These samples were instrumental as controls in our *ACS Nano* paper for determining the mechanisms of inter-fiber energy transfer because the fibers are so well separated in these samples that inter-fiber energy transfer is suppressed. Moreover we discovered that the pores can be back-filled with solution-processable C₆₀-derivative electron acceptors and the films then partially re-condensed to create bulk heterojunctions with improved power conversion efficiency compared to planar devices (published in *Small*).

2. Dynamics of CO₂ reduction via solvated electrons

During the last year we identified conditions necessary to achieve highly selective photochemical reduction of CO₂ to CO in aqueous media. One of the most striking aspects of this work is that by using modest internal pressures in the photolysis cell, we are able to almost completely eliminate the electrochemical reduction of H⁺ to the hydrogen atom $H^+ + e^-_{(aq)} \rightarrow H^{\bullet}_{(aq)}$. This is an important result, because in virtually all previous studies of CO₂ reduction in aqueous media, formation of H₂ is the predominant product. This remarkable selectivity for CO₂ reduction is due in large part to the kinetic rates of electron-transfer reactions: Although the rate constant for $H^+ + e^-_{(aq)} \rightarrow H^{\bullet}_{(aq)}$ is large, by using modest (10-20 atmospheres) for pCO₂, the overall rate for $CO_2 + e^-_{(aq)} \rightarrow CO_2^{\bullet-}_{(aq)}$ can be made to exceed that for $H^+ + e^-_{(aq)} \rightarrow H^{\bullet}_{(aq)}$, leading to >95% selectivity for CO₂ reduction even at pH=3.5. A second critical parameter is having a mechanism for removing valence-band holes before they induce oxidation

of the diamond (which also forms CO). Every electron emitted from diamond must be accompanied by an oxidation process to maintain charge neutrality; however, diamond's shallow valence band position yields only low rates for the necessary oxidation processes. We found two approaches to solving this problem: (1) using a secondary Pt electrode to oxidize water, or (2) including a hole scavenger (such as sulfite) in the reaction mixture. The use of a small (~ -0.5 V) potential to the diamond also helps. We confirmed selectivity of reaction and the absence of competing oxidation of diamond itself using isotope labeling studies. Our experiments showed that gas-phase $^{13}\text{CO}_2$ yielded exclusively ^{13}CO with no significant ^{12}CO .

While our initial work indicated that nanodiamond was particularly prone to oxidation, we also found that inexpensive diamond nanopowder (<125 nm) can achieve similarly high selectivity provided that a suitable hole scavenger is used. Product distributions were measured by FTIR and NMR spectroscopy. Illumination-induced changes in diamond nanoparticle oxidation state and aggregation state were also performed. Ultimately this work demonstrates that inexpensive diamond nanopowder can be an effective homogeneous photocatalyst for CO_2 reduction.

We also provided diamond electrodes for investigations by Dr. Tim Berto in Prof. John Berry's group investigating the electrolyte composition on the electrochemical reduction of CO_2 . That work showed that, contrary to reports published by other groups, tetraalkylammonium ions are not catalysts for CO_2 reduction.

Complete List of publications supported in-full or in-part by this grant

Randy D. Mehlenbacher, Thomas J. McDonough, Maksim Grechko¹, Meng-Yin Wu, Michael S. Arnold, Martin T. Zanni, Energy Transfer Pathways in Semiconducting Carbon Nanotubes Revealed using Two Dimensional White-Light Spectroscopy, *Nature Communications*, 6, 6732 (2015).

Shea MJ, Mehlenbacher RD, Zanni MT, Arnold MS. 2014. Experimental Measurement of the Binding Configuration and Coverage of Chirality-sorting Polyfluorenes on Carbon Nanotubes. *J. Phys. Chem. Lett.* 5, 3742 (2014)

Shu Yao, Rose Ruther, Linghong Zhang, Ryan Franking, Robert Hamers, and John Berry, J. "Covalent attachment of catalyst molecules to conductive diamond: CO_2 reduction using "smart" electrodes", *J. Am. Chem. Soc.* 2012, 134, 15632-15635.

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Ye Y, Bindl DJ, Jacobberger RM, Wu M-Y, Singha Roy S, Arnold MS, Semiconducting Carbon Nanotube Aerogel Bulk Heterojunction Solar Cells, *Small* 10 (16), pp 3299-3306 (2014).

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Linghong Zhang, Di Zhu, Gilbert M. Nathanson, and Robert J. Hamers, "Selective Photochemical Reduction of Aqueous CO₂ to CO by Solvated Electrons", *Angewandte Chemie* **2014**, 53, 9746+.

Linghong Zhang and Robert J. Hamers, "Achieving Selective CO₂ Photoreduction to CO with Diamond Nanoparticles", in preparation (manuscript complete, nearly ready for submission).

Archival Publications (published) **during reporting period:**

Randy D. Mehlenbacher, Thomas J. McDonough, Maksim Grechko¹, Meng-Yin Wu, Michael S. Arnold, Martin T. Zanni, Energy Transfer Pathways in Semiconducting Carbon Nanotubes Revealed using Two Dimensional White-Light Spectroscopy, *Nature Communications*, 6, 6732 (2015).

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Martin Zanni, Michael Arnold, and Robert Hamers

Program Manager**The AFOSR Program Manager currently assigned to the award**

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